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Application for Patent (A)

July 15, 1975
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TO: Director of the Japanese Patent Office

1. Name of Invention:
Manufacturing Method for Processed Edible Oil and Fat Products

2. Inventor:
Toyoaki YONEDA, 400-108 Joza, Sakura City, Chiba-ken
(Three additional names, attached)

3. Patent Applicant:
11, 1-chome, Minamimoto-cho, Higashi-ku, Osaka
(300) Teijin, KK
President and CEO, Shinzo OYA

4. Patent Attorney:
1-1, 2-chome, Uchisaiwai-cho, Chiyoda-ku, Tokyo
(Iino Building)
Teijin, KK
Sumihiro MAEDA, Patent Attorney (7726)
Contact: (506)4481 Isao/Takeshi Takayama

/seal/

5. Attachments:
(1) Detailed Explanation of Invention 1 copy
(2) Power of Attorney 1 copy
(3) Copy of Application for Patent 1 copy

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Detailed Explanation of the Invention

1. NAME OF THE INVENTION

Manufacturing Method for Processed Edible Oil and Fat Products

2. CLAIMS

Manufacturing process for processed edible oil and fat products in which fatty-acid and glycerin triglyceride with cyclodextrin inclusion compounds serve as emulsifying agents.

3. DETAILED DESCRIPTION OF THE INVENTION

The present Invention pertains to a manufacturing method for edible processed oil and fat products. More specifically, it pertains to the use of a triglyceride (fatty-acid and glycerin) dextrin inclusion compounds as emulsifiers. In other words, this Invention pertains to a manufacturing process which utilizes a new type of emulsifier for emulsifying edible processed oils and fats.

Oils and fats are said to be one of the three primary food groups of man. In addition to their use as a means for grilling and deep frying food products, they also serve as an ingredient used directly in foods, i.e., butter, margarine, shortening, mayonnaise, etc.

As oils and fats are inherently difficult to digest, previously mentioned emulsification and flavor enhancers have been used to improve edibility. In the case of highly unsaturated oils and fats, the addition of hydrogen has been used as a hardener as well as a means for masking undesirable odors, thereby greatly enhancing palatability.

Surfactants have also been used to emulsify oils and fats. Although they are considered to be permissible as food additives, their amounts must be kept to a minimum however. For instance, the maximum fatty-acid ester level of cane sugar is HLB13, such that higher HLB levels have not been seen. This renders O/W type immersion stability very difficult when water content is minimized, conversely, electrolysis limits pH levels with the addition of water.

The author's findings from in-depth studies on providing the optimum emulsifier for use in processing O/W type emulsified food products indicate that O/W type immersion is especially stable when water content is minimal. For instance, reports of attempts to prevent cholesterol

precipitation have shown excellent results when necessary fatty acids in many popular rice-bran and safflower oils (containing unsaturated fatty-acids) are emulsified without the addition of water. The present Invention achieves this ideal as well.

That is to say, this Invention is a unique manufacturing method for processed edible oils and fat products, the emulsifier of which is a triglyceride (fatty-acid and glycerin) type cyclodextrin inclusion compound.

Glycerides of fatty acids and glycerin, as mentioned in this Invention, are the primary constituents in the fats and oils which occur naturally in a wide variety of plants and animals. Generally speaking, they are defined as those fats ranging from 4-carbon lactic acid to the 24-carbon triglycerides of fatty-acids and glycerins. As such, it is easy to understand how saturated fatty-acids such as the 16-carbon palmitate and 18-carbon stearic acid, as well as unsaturated fatty-acids such as linoleic acid, linolenic acid, and triglycerides of glycerin, etc. are considered representative of fatty-acids.

Although cyclodextrins, and in particular Schardinger dextrans, are well known chemical compounds, this Invention concerns itself with α -cyclodextrins (cyclohexa-aminose), β -cyclodextrins (cyclohepta-aminose), γ -cyclodextrins (cycloocta-aminose), etc. and or the application of various compounds of their key internal constituents. For example, as disclosed in unexamined Japanese Patent SHO 46-2380 (1971), it is possible to manufacture Pachels-Maserans aminose in liquefied starch with less than DE15. Under such processing conditions, for example, the primary ingredients in α -cyclodextrin and β -cyclodextrin are obtainable. More often than not however, a disparate variety of cyclodextrin compounds are yielded.

Simply stated, the present Invention and its use of the above-mentioned triglyceride (fatty-acid and glycerin) dextrin inclusion compounds entails a production process whereby triglycerides and cyclodextrin are mixed and thoroughly blended in existing water. In other words, certain amounts of solution are naturally formed when certain amounts of water are mixed with certain amounts of cyclodextrin to form a slurry-type substance.

In order to clarify this further, the following example describes the use of inclusion compounds in a manufacturing process in which the glycerides of fatty-acid and glycerin are in the form of rice-bran oil spread.

Ten parts (by weight) of cyclodextrin to 30 parts of water are thoroughly blended into a paste to which 20 parts (by weight) of rice bran oil spread are added and thoroughly blended once again. The more vigorous the mixing process, the better the results, such that the use of a homomixer is recommended in order to achieve the ideal composition. As described in the above-mentioned operation, the continuous processing of the inclusion compound and/or simultaneous emulsification of the triglycerides provides the distinct advantage of obviating the isolation of the inclusion compound.

As for the above-mentioned manufacturing processes in which the inclusion compounds are isolated, processed ethyl solvents are easily dissolved when the appropriate amounts of water, oils and fats are thoroughly blended. Moreover, appropriate procedures related to decantation, extraction, filtering, centrifugal separation, etc. must be applied accordingly.

It is thought that the product derived by the above process is the result of the formation of the inclusion compound as a part of the fatty-acids/glycerin triglyceride molecule chain, such that the inclusion exists in the cyclodextrin ring. The reason for the ease at which the inclusion compound is formed so easily, is due to the abundance of oleophylic hydrogen atoms on the cyclodextrin rings. As there is a relative abundance of hydrogen radicals on the outside of the hydrophylic rings, some or all of the oleophylic molecules on the inside of the rings are thus readily absorbed.

The derivatives created by the above-mentioned methods of this Invention and their inclusion compounds, have been verified by the inventor by means of actual experiments.

The derivatives obtained after the available water in cyclodextrin and triglyceride (fatty acid and glycerin) was appropriately blended were then depleted of excess water, after which the non-inclusion cyclodextrin was extracted. The non-inclusion triglyceride was then extracted after residual ether, acetone and other solutions were removed. After drying, the product was then heated until becoming a blended solution (medium) with a methanol to acetone ratio of 50:50 (by weight), from which the triglyceride was extracted. After weighing the product, the molecular ratio of dried cyclodextrin to triglyceride may be calculated.

Emulsification was shown to be significant when results indicated a ratio of 1:1 to 3:1. Given the above-mentioned drying process and the indication of specific mol ratios, the cyclodextrin inclusion of triglyceride as a guest compound was ascertained. When this Invention is applied to

various oil and fat products for which emulsification is necessary, the question arises as to the ideal approach to be taken, in particular, for processing margarines, mayonnaise, salad dressings, cream cheeses, flower pastes, chocolate pastes, soft ice cream mixes, etc.

The variety of applications for inclusion compounds depends on the type of food product in question, therefore it is best to have a specific application in mind when using any of the common surfactants, such as mono-fatty-acid esters of glycerin, etc. The inclusion of cyclodextrins and triglycerides is a relatively simple matter when the presence of water in the inclusion compound is minimal, as the minimum water obviates the isolation of the inclusion compound as discussed in the above methods. As for the manufacturing of emulsified food products, it is the presence of hydrogen in the cyclodextrin and the proper mixing of ingredients which successfully brings about the desired results. In actual practice, heretofore discussed methods are ideal for industrial application. In the practical examples below, inclusion compounds are not regarded as "additives" per se, instead, they are regarded as being the essential constituent comprising the system itself; their function as such should help simplify their application in this regard.

The amount of inclusion compound to be used is dependent on the amount of cyclodextrin to be used. In terms of the stated method, if the water, oils and fats in the processed food ranges between 0.5% to 10% by weight, the ideal proportion would be 2 to 6% by weight, respectively. According to the inventor's research however, when the ratio of oils and fats to water exceeds 1:1 by weight, then the ideal proportion of the cyclodextrin should ideally be greater than that stated above, for example, more than 3% by weight, or better yet, more than 4% by weight.

In addition to their use as emulsifiers as described in this Invention, triglycerides of fatty-acid and glycerin containing cyclodextrin inclusion compounds serve yet another function. At this same time, for example, it is already known that they may be used with emulsifiers such as fatty-acid esters of cane sugar, soy lecithin, egg yolks, etc., so naturally their combined applications are obvious. As for their combined use with emulsifiers in this manner, there is particular interest in their application with non-protein milkless cream bases as a food texturizer. This unique characteristic manifests a tremendous added value of their use in improving the quality of food products.

Listed below are detailed descriptions of actual examples of the application of this Invention. It must be emphasized however, that the following examples do not limit the scope of this Invention in any way, instead the purpose is to describe the essential and unique aspects of the Invention.

Example 1 (Margarine type spread)

β -cyclodextrin	4 parts
Water	27 parts
Rice oil, prepared (rice oil: safflower oil = 7:3, by weight)	60 parts
Sugar, granulated	10 parts
Fermented milk	5 parts
Xanthan gum	0.01 parts
Flavorings (Mirufa M—Manufactured by Takasago (KK)	0.3 parts
Butter flavor	0.08 parts

After adding 27 parts of water to 4 parts of β -cyclodextrin, blend in a homomixer to disperse the solution. Add 10 parts of prepared rice oil and mix for approximately 10 minutes at which time the cyclodextrin inclusion compounds in the prepared rice oil will emerge.

Next, add in 10 parts of granulated sugar, 5 parts of fermented milk, and 0.01 parts xanthan gum; mix for approximately 15 minutes. To this mixture, add 50 parts of prepared rice oil and mix for approximately 20 minutes. As the mixture is blended, it will also be emulsified. Finally, after adding in 0.3 parts of Mirufa M and 0.08 parts butter flavor and blending evenly, the end product will be a sweet margarine type spread.

Fill a container with the product made from the above-mentioned method and after storing at -10°C for one week, restore the product to 20°C for 12 hours. If the emulsified product that has been chilled and then thawed visually appears similar to that of the original (unchilled and unthawed) product, then results may be considered excellent.

Example 2 (Margarine type spread)

β -cyclodextrin	4 parts
Water	27 parts
Rice oil, prepared (Same as that used in Example 1)	60 parts
Fermented milk	5 parts
Table salt	1 part
Xanthan gum	0.1 parts
Mirufa M	0.3 parts
Butter flavor	0.1 parts

After mixing 4 parts of β -cyclodextrin, 25 parts of water, 1 part of table salt, and 0.1 part of xanthan gum in a homomixer until it is liquefied and then dispersed, add 7 parts of prepared rice oil and mix continuously for approximately 15 minutes.

Next, add in 5 parts of fermented milk and mix for approximately 10 minutes, and then add in 53 parts of prepared rice oil and mix for approximately 20 minutes until it is emulsified. Finally, after adding in 0.3 parts of Mirufa M and 0.1 parts butter flavor and blending evenly, the end product will be a salty margarine type spread. The stability of this product may be determined according to the same method used in Example 1, such that if the (chilled and thawed) product visually appears similar to that of the original (unchilled and unthawed) product, then results may be considered excellent.

Example 3 (Milkless Cream Base)

β -cyclodextrin	5 parts
Soybean shirashime oil (either vegetable or animal oil may be used)	45 parts
Water	50 parts
Lecithin	0.4 parts
Monoglyceride	0.1 parts
Milk-free butter	0.1 parts

Mix 50 parts of water, the β -cyclodextrin, and 0.1 parts of monoglyceride in a homomixer until a well blended water-base is created. In a separate process, mix 45 parts of Soybean shirashime oil and 0.4 parts of lecithin in a homomixer until blended and an oil-base is created.

After gradually add the oil-base into the above-mentioned water-base (or vice versa is also acceptable), place the mixture into a high pressure homogenizer and mix until a uniformly blended milkless cream base develops. (Ideal homogenizer pressure range: 30~200 Kg/cm² to 50~100 Kg/cm² [Kilogram/square centimeter/liter]).

In order to use this milkless cream base for making whipping cream, add in 10 to 20 parts of sugar and ideally, add milk protein as necessary.

Example 4 (Eggless Mayonnaise)

β -cyclodextrin	4 parts
Salad oil	70 parts
Apple vinegar (in general, fresh vinegar may be used)	9 parts
Sugar, granulated	2 parts
Salt, table	1.2 parts
Karashi [Japanese mustard], powder	0.8 parts
Koshō [Japanese pepper], powder	0.1 parts
Paprika, powder	0.1 parts
Water	1.5 parts

After mixing 4 parts of β -cyclodextrin, 2 parts of granulated sugar, 1.2 parts of table salt, 9 parts of apple vinegar, and 1.5 parts of water in a homogenizer, add in 10 parts of salad oil and mix until thoroughly blended. Next, add 0.8 parts of powdered Japanese mustard, 0.1 parts of Japanese pepper, and 0.1 parts of paprika power and mix once again until thoroughly blended. Lastly, add the remaining 60 parts of salad oil and thoroughly mix in similar fashion until a uniformly blended eggless mayonnaise is produced.

Example 5 (Salad Dressing)

β -cyclodextrin	3 parts
Salad oil	35 parts
Starch, powdered	5 parts
Sugar, granulated	20 parts
Salt, table	2 parts
Apple vinegar	15 parts
Water	53 parts
Spices (to taste)	0.5 parts

Under heat, mix 5 parts of starch powder, 20 parts of granulated sugar, 2 parts of table salt, and 20 parts of water to make a well blended starchy paste. In a separate process, mix 3 parts of β -cyclodextrin, 15 parts of apple vinegar, and 33 parts of water and blend thoroughly. Next, mix in 10 parts of salad oil and blend thoroughly.

Gradually add the ingredients to the above-mentioned starchy paste base while mixing in order to obtain a thoroughly blended mixture. Next, add in 25 parts of salad oil and thoroughly blend in similar manner. Finally, add the 0.5 parts of flavorings until a thoroughly blended salad dressing is produced.

Place the product in a transparent container and store in a cold environment for one month and observe whether the product separates. Instances in which products separate should be disregarded.

Example 6 (Soft Cream Mix)

β -cyclodextrin	5 parts
Hydrogenated oil	45 parts
Soy lecithin	0.4 parts
Monoglyceride	0.1 parts
Starch, powdered (blended mix)	0.5 parts
Nonfat milk	30 parts
Water	50 parts
Ice cream flavoring(s)	0.1 parts

After mixing 50 parts of water, 5 parts of β -cyclodextrin, 0.1 parts of monoglyceride, 0.5 parts of powdered starch (blended mix), and 30 parts of nonfat milk in a homogenizer until a thoroughly blended water base is produced.

In a separate process, heat 45 parts of hydrogenated oil until dissolved, add in 0.4 parts of lecithin and mix in a homogenizer in a similar fashion until thoroughly blended oil base is produced.

After gradually add the oil-base into the above-mentioned water-base (or vice versa is also acceptable), place the mixture into a high pressure homogenizer and mix until a uniformly blended soft cream base develops. (Ideal homogenizer pressure range: 30~200 Kg/cml. Pressure setting of 50~100 Kg/cml is particularly ideal.

As a test, store the soft cream base product for one month at 5°C. If the product does not separate or manifest any changes in quality, it may be considered to be of excellent quality.

Example 7 (Flour Paste)

β -cyclodextrin	1 part
Salad oil (either vegetable or animal oil may be used)	7 parts
Sugar, granulated	16 parts
Sugar, glucose	9 parts
Starch syrup	10 parts
Corn starch	1 parts
Processed starch	9 parts
Nonfat milk	1.2 parts
Egg yolk, powdered	0.2 parts
Table salt	0.05 parts
Water	40 parts
Sodium biphosphate	0.01 parts
Coloring	to taste
Flavorings/spices	to taste

After dissolving 1 part of β -cyclodextrin and 8 parts of granulated sugar in 10 parts of water, mix in 10 parts of the liquid vegetable or animal fat (oil) until thoroughly blended. (Cream base).

In a separate process, add 8 parts of granulated sugar, 9 parts of glucose sugar, 10 parts of starch syrup, 1 part of corn starch, 9 parts of processed starch, 1.2 parts of nonfat milk, 0.2 parts of powdered egg yolk, 0.05 parts of table salt, and 0.01 parts of sodium biphosphate to 30 parts of water. Mix thoroughly while heating. After cooling, mix thoroughly with the above-mentioned cream base. Next, add in the desired coloring and spices as a flour paste.

Unlike other flour pastes, the merit of this product is that it does not dry out when either stored or placed in a pan into which fillings are added.

Applicant

Teijin, KK

Agent and Patent Attorney

Sumihiro MAEDA

6. Inventors not listed above:

Masayuki KITANAKA

1319-79-405, Makita-cho, Takatsuki City, Osaka-fu

Atsuyuki SHIMA

1-18-4 Tamadaira, Hino City, Tokyo

Naohisa SAITO

1-18-4 Tamadaira, Hino City, Tokyo

Amendment to Patent Filing

August 14, 1975

TO: The Director of the Japanese Patent Office

1. Designation of Case

Patent Application Filing Number: SHO-50-86429-GO

2. Name of Invention

Manufacturing Method for Processed Edible Oil and Fat Products

3. Party Making the Amendment

Connection with Case Patent Applicant

**11, 1-chome, Minamimoto-cho, Higashi-ku, Osaka
(300) Teijin, KK**

4. Patent Agent (Attorney)

**1-1, 2-chome, Uchisaiwai-cho, Chiyoda-ku, Tokyo
(Iino Building)**

Teijin, KK

Sumihiro MAEDA, Patent Attorney (7726)

Contact: (506)4481 Isao/Takeshi Takayama /seal/

5. Object of Amendment

**"Detailed Description of the Invention" section of the
Detailed Explanation of the Invention.**

/seal

/stamp August 14, 1975, 2nd Office, Applications, JPO

6. Amendments

- (1) Section entitled "Detailed Explanation of the Invention", page 7, line 13 [of original Japanese document]: correct "bathing agent" to read "solution" [obvious typographical error].**
- (2) Same section, page 8, line 1: amend "1:1 per 3:1" to read "1:1~3:1".**
- (3) Same section, page 9, line 1: amend "within it" to read "inside it".**
- (4) Same section, page 9, line 2: amend "water related" to read "water system".**
- (5) Same section, page 9, line 8: amend "primary constituent" to read "formation".**
- (6) Same section, page 13, lines 4~5: amend "the manufacturing method in Example 1" to read "the product produced by the method in Example 1".**

Respectfully submitted

Amendments made to Name(s) of Patent Applicant

2000 yen

(2,400 yen)

March 16, 1976

TO: The Director of the Japanese Patent Office

1. Designation of Case

2. Name of Invention

Manufacturing Method for Processed Edible Oil and Fat Products

3. Party Making the Amendment

(300) Teijin, KK

11, 1-chome, Minamimoto-cho, Higashi-ku, Osaka

Representative: Shinzo OYA

4. New Holding Participant

28-15, 3-chome, Mitsuya Minami, Yodogawa-ku, Osaka

Ripon Foods, KK

Representative: Oichi IKADA

5. Patent Agent (Attorney)

1-1, 2-chome, Uchisaiwai-cho, Chiyoda-ku, Tokyo

(Iino Building)

Teijin, KK

Sumihiro MAEDA, Patent Attorney (7726)

/seal

6. List of Appended Documents

(1) Joint-Holding Agreement 1 copy

(2) Power of Attorney 1 copy

/JPO date stamp, March 16, 1976